

BATTELLE
WORK/QUALITY ASSURANCE PROJECT PLAN

1.0 GENERAL PROJECT INFORMATION

Project Title: Montrose Settlement Restoration Program Tissue Analysis
Project Number: G004778
Client: Dr. Ann Jones
Industrial Economics, Inc.
2067 Massachusetts Avenue
Cambridge, MA 02140
Phone: (617) 354-0074
QAPP Effective Date: February 22, 2005 (Rev. 2 and project initiation)
Version Number: 2005-Rev 4
Project Manager: Robert Jr.
Prepared By: Robert Lizotte, Jr.  Date: April 27, 2005
Reviewed By: William Steinhauer  Date: April 28, 2005
Deliverable Due Date: To be determined

2.0 SCOPE OF WORK

Objective and General Information

The objective of this project is to analyze and determine the concentrations of selected PCB congeners and pesticides (Attachment 1. Test Code) in fish tissue samples. In addition the total extractable organics (TEO) and % moisture content will be determined and reported. All results will be reported on a wet weight basis and surrogate corrected.

2.1 TECHNICAL APPROACH

2.1.1 Sample Receipt, Storage, and Handling

The samples have been frozen and logged into the LIMS and given unique Battelle IDs. The applicable storage and holding time information is as presented in Table 1:

Table 1: Sample Storage and Holding Times

Sample Type	Storage	Holding Times
Tissue	Frozen (-10 °C or less)	NA

Unexpended tissue samples will be held and returned to the client at the end of the project. Sample extracts will be held until after the delivery of the final data (90 days) and disposed of at the direction of the project manager. The electronic data generated by the instruments and all hard-copy data will be archived for a period of 10 years, unless otherwise specified by the client in writing.

2.1.2 Sample Preparation

Most samples were previously homogenized; however, there will be some samples that need to be filleted and homogenized.

2.1.2.1 Fish Measurement

Only previously un-filleted fish will need to be measured.

Each fish has been measured in the field to allow selection of certain size classes for analysis. In the laboratory, each fish selected for resection and analysis will be measured again and weighed. Total length (to 1 mm) and weight (to 0.1 gram for small fish and 1 gram for fish greater than 100 grams) of each fish will be measured and recorded, along with the identification code. If there is a significant discrepancy in the total length (greater than 10 %) the sample will be flagged and the IEC PM notified due to the indication of a potentially mis-recorded fish.

See Attachment 2 for fish measurement techniques. All fish will be measured and weighed before filleting (see SOP Montrose 001-01 for details and log record for weight and measurement).

2.1.2.2 Fish Filleting

Fillet samples will be removed from each fish for analysis. Fish will be scaled and filleted in the laboratory following Montrose SOP 001-01. A fillet will be taken from the whole of one side of the partially frozen fish, directly behind the pectoral fin (See Attachment 3). The fillet will be carefully cleaned to remove skin and fatty tissue. Any trimmings will be retained with the remainder of the fish. These will be resealed in a plastic bag and properly labeled. The belly flap will not be included with the fillet, but will remain with the whole body.

If homogenization is not completed at the same time, the fillet sample will be placed on a tared sheet of aluminum foil and weighed. The fillet will then be wrapped in the foil and stored in a plastic bag and labeled with the Battelle **III** number.

2.1.2.3 Homogenization of Fillet Samples

Follow the same decontamination precautions as when performing the dissections. Upon completion, the homogenate for each sample will be kept, stored frozen in tared, certified clean glass jars with a polytetrafluoroethylene (PTFE) lid. The sample number will be amended to indicate the type of sample (e.g., WC-003-F for homogenate of a white croaker fillet).

Sample duplicates will be run once with each batch, to ensure adequate homogenization. If the duplicate results do not meet the specified measurement performance criteria, the batch will be re-homogenized and re-sampled.

Rinsate blanks will not be performed with this work. Previous rinsate blanks have demonstrated the cleanliness of our procedure.

IEc will provide direction on the sample make up of each extraction batch. The samples will be analyzed in batches of no more than 15 field samples of the same sample matrix. The quality control samples listed in Table 2.

Table 2: Quality Control Samples

<i>QC sample Type</i>	<i>Required (check)</i>	<i>Comments</i>
Reagent Blank	√	Hydromatrix
Method Blank	√	Talapia homogenate
LCS (mid-level)	√ (Every other batch)	Talapia homogenate
LCS (low-level)	√ (Every other batch)	Talapia homogenate
MS	√	
Sample Duplicate	√	Use the sample with the most tissue from that batch
SRM	√	SRM1946. Use about 5g (do not thaw the SRM)

2.1.2.4 Tissue Extraction/Preparation

Reference the following SOPs for sample extraction and processing:

SOP 5-307-03, *Soil/sediment and tissue extraction for semi-volatile contaminant analysis using the accelerated solvent extractor* (note that the incorrect date is in the header)

- DCM will be used as the extraction solvent

SOP 5-191-07, *HPLC (GPC) cleanup of sample extracts for semi-volatile organic pollutants* (note that there is no date is in the header)

SOP 5-327-01, *Florisil Cleanup of Environmental Sample Extracts*

Extract a 5-g samples by accelerated solvent extraction following Battelle SOP 5-307. Extracts will be dried, concentrated following the Kuderna-Danish procedure, filtered, and a total extractable organic (TEO) weight will be determined following procedures in this SOP. Perform size-exclusion HPLC clean-up following procedures in SOP 5-191, then perform Florisil cleanup following SOP 5-327. SIS and LCSIMS spiking levels are presented in Table 3. Internal standard spiking levels are presented in Table 4.

The final PIV is 500 µL.

Table 3: SIS and LCS/MS Spiking Level

Standard Type	Standard Contents	Spike Amount (ng)	Standard ID (or equivalent)	Volume (µL)	Comments
Surrogate Internal Standards (SIS)	PCB 36,192	~ 100 ng	GF94 or equivalent	100	All samples
Low level LCS	Target PestJPCB congeners	~50 ng pest/ 12.5 ng PCB congener	GD80 or equivalent	25 µL	Low level LCS only
Mid level LCS	Target PestJPCB congeners	~400ng pest/ 100 ng PCB congener	GD80 or equivalent	200	Mid level LCS only

Table 4: IS Spiking Level

Standard Type	Standard Contents	Spike Amount (ng)	Standard ID (or equivalent)	Volume (µL)	Comments
Internal Standards (IS)	PCB 96	~ 50 ng	GF95 or equivalent	50	All samples

2.1.3 Instrumental Analysis - Pesticide/PCB Congener/PCB LOC Analysis by GC/MS

Follow procedures presented in SOP Montrose 002-05. The additional directions apply:

- Report and flag all data above the MDL according to Battelle SOP 7-029 *Preparation, Analysis, and Reporting Quality Control Data in the Chemistry Laboratory*.
- Report data to 3 significant figures.
- Evaluate the data against the measurement quality objectives (MQOs) presented in Attachment 4.
- Data qualifier definitions are presented as Attachment 5.
- The procedural/method blank concentrations will be reported on a wet weight concentration basis, using the average (5g) field sample weight (tissue).
- Sample-specific MDLs are required. MDL adjustments will be made for sample weights and sample dilutions. Insert MDLs in the value field for non-detects and flagged with a U.
- Only the highest or highest two calibration points may be dropped from the calibration curve.

2.2 DELIVERABLES

A full data package will be delivered. Table 5 presents the contents of each data package. An electronic deliverable is required. See Attachment 6 for electronic deliverable format.

Table 5. Data Package Deliverables
Case narrative
Cross reference of field sample no., laboratory sample no., and analytical batch
Chain-of-Custody form (including sample receipt checklist)
Sample duplicate results and relative percent difference (RPD) Values
Reference material results and performance criteria assessment
Internal standard recoveries (format at laboratory discretion)
Initial calibration for single component analytes, retention time windows
Initial calibration for single component analytes, response factors.
Calibration verification, including end-of-run verification.
Size exclusion HPLC check
Chromatograms and instrument printouts for each sample, blank, and standard
Sample calculation
Blank spike results
Surrogates recovery
Instrument tune
Matrix spike results and recoveries
Quantitation report
Copies of Run Logs
Results summary for each sample and blank
Copies of Sample Preparation Work Sheets

3.0 QUALITY

3.1 QUALITY ASSURANCE

The Quality Assurance Unit (QAU) at Battelle will remain independent of all work activities pertaining to this project. The QAU will monitor the Battelle components of the project according to existing Battelle SOPs to ensure the accuracy, integrity, and completeness of the data. Additionally, the QAU will monitor the project activities to ensure consistency with the applicable requirements described in this QAPP. The QAU scope includes system inspections, data audits, and reviews of documents and deliverables.

3.2 QUALITY CONTROL

Project staff will be responsible for ensuring that sample tracking, sample preparation, and analytical instrument operation all meet the quality control criteria detailed in the applicable analytical SOPs. The type and frequency of analysis of QC samples is specified in Section 2.

All data will be audited against the SOP requirements and the MQO presented in Attachment 4. Analytical results that do not meet the listed DQOs will be submitted to and reviewed with the Project Manager in a timely manner for assessment of the potential impact of the results. Affected samples may be reanalyzed at the request of the Project Manager, following discussion with IEc. Quality control sample data that are accepted outside these criteria will be indicated with the appropriate data qualifier, and the rationale for accepting the analysis will be documented in the project files.

3.3 DOCUMENTATION

All data will be initially recorded either (1) electronically onto computer storage media using the laboratory LIMS systems or (2) manually into laboratory notebooks or onto established data forms. All notes and records, including data, will be recorded promptly, directly, legibly, in ink. If corrections to hand-recorded data are required, a single line will be drawn through the entry, which will be initialed, dated, and justified. Corrections to electronically captured data will be documented within the LIMS system, and the instrument electronic change-control tracking system, or on a hard-copy of the data if these options are not available to a laboratory. Completed data forms or other types of hand-entered data will be signed and dated by the individual entering the data.

4.0 ORGANIZATION AND COMMUNICATION

The Battelle project team and responsibilities are presented in Table 6.

Table 6: Project Team and Roles

Staff Member	Role
Robert Lizotte	Project Manager
Roxanne Brackett/Denise Thompson	Sample Preparation
Susan O'NeillMichael Mitchell	GCIMS Analysis
Jeffery Newell	Sample Custody Officer
Rosanna Buhl	Quality Systems Manager

5.0 SCHEDULE

Laboratory work will begin upon approval contract award, expected early March 2005.

6.0 BUDGET

The labor budgets associated with activities presented in this work plan should be charged to G004778-2000. Labor budget expectations will be communicated separately.

7.0 STAFF DEVELOPMENT

Staff development expectations will be communicated separately.

ATTACHMENTS

- Attachment 1: Test Codes
- Attachment 2. Fish Measurement Technique
- Attachment 3. Fish Filleting Technique
- Attachment 4. Method Quality Objectives
- Attachment 5: EDD Specifications

Sample Custody forms are not attached to this QAPP. The appropriate custodies for the appropriate samples in each batch will be included in the package.

ATTACHMENT 1
TEST CODE

Project Test Code Summary Report

Lab Name: MS_PestPCB

SOP Reference: M002 - Identification and Quantification of Polychlorinated Biphenyl Congeners (PCBs), Chlorinated Pesticides and PCB Homologues by Gas Chromatography/Mas Spectroscopy in the Select Ion Monitoring (SIM) Mode.

Description: MS_PestPCB

Matrix: T - Tissue Samples, like fish or animal, prepared and analyzed under the same class of detection limits.

Report Name: MS_PestPCB

Detection Limit Study: MDL-2004-MSRP **Instrument:** Mass Spectrometer (GCMS)

DQO Criteria: Montrose

Standard Report: Standard Result Report

Result Units:	<u>ng/g</u>	Unit Conversion:	<u>(none)</u>	Holding Times (days)		
Weight Basis:	<u>WET</u>	Result Format:	<u>Significant Figures</u>	Sample:	<u>14</u>	DL_Flag: <u>U</u>
Standard Basis:	<u>SIS</u>	# of Figures/Digits:	<u>3</u>	Extract:	<u>40</u>	RL_Flag: <u>J</u>
Oil Weight Basis:	<u>No</u>	Oil Weight Source:	<u>Oil Weight</u>	Frozen:	<u>40</u>	PB_Flag: <u>B</u>
U-Value Substitution:	<u>ND=MDL</u>			HT_Flag:	<u>T</u>	DIL_Flag: <u>D</u>

No:	Laboratory Analyte Name:	Report Name:	Type	RIS	SIS	DL	Units:
1	4,4'-000	4,4'-000	T	Cl5(96)	Cl7(192)	0.943606539	ng
2	2,4'-000	2,4'-000	T	Cl5(96)	Cl7(192)	0.735312065	ng
3	4,4'-00E	4,4'-00E		Cl5(96)	Cl7(192)	1.559808561	ng
4	2,4'-00E	2,4'-00E	T	Cl5(96)	Cl7(192)	0.231435287	ng
5	4,4'-00T	4,4'-00T		Cl7(186)	Cl7(192)	0.697601166	ng
6	2,4'-00T	2,4'-00T	T	Cl5(96)	Cl7(192)	0.857948222	ng
7	a-chlordane	a-chlordane	T	Cl5(96)	Cl7(192)	0.905398983	ng
8	g-chlordane	g-chlordane	T	Cl5(96)	Cl7(192)	0.633940948	ng
9	cis-nonachlor	cis-nonachlor	T	Cl5(96)	Cl7(192)	0.484781246	ng
10	trans-nonachlor	trans-nonachlor	T	Cl5(96)	Cl7(192)	0.663814711	ng

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Project Test Code Summary Report

Lab Name: MS PestPCB

No:	Laboratory Analyte Name:	Report Name:	Type	RIS	SIS	DL	Units:
11	dieldrin	dieldrin	T	CI5(96)	CI7(192)	1.964363576	ng
12	oxychlorane		T	CI5(96)	CI7(192)	1.391262621	ng
13	CI2(8)	CI2(8)	T	CI5(96)	CI3(36)	0.323251467	ng
14	CI3(18)	CI3(18)	T	CI5(96)	CI3(36)	0.332540139	ng
15	CI3(28)	CI3(28)	T	CI5(96)	CI3(36)	0.347694584	ng
16	CI3(31)	CI3(31)	T	CI5(96)	CI3(36)	0.220752048	ng
17	CI3(37)	CI3(37)	T	CI5(96)	CI7(192)	1.497526167	ng
18		CI4(44)	T	CI5(96)	CI7(192)	0.437292618	rig
19	CI4(49)	CI4(49)	T	CI5(96)	CI7(192)	0.298484042	ng
20	CI4(52)	CI4(52)	T	CI5(96)	CI7(192)	0.742150065	ng
21	CI4(66)	CI4(66)	T	CI5(96)	CI7(192)	0.231629215	ng
22	CI4(70)	CI4(70)	T	CI5(96)	CI7(192)	0.506478576	ng
23	CI4(74)	CI4(74)	T	CI5(96)	CI7(192)	0.207677769	ng
24	CI4(77)	CI4(77)	T	CI5(96)	CI7(192)	0.223980768	ng
25	CI4(81)	CI4(81)	T	CI5(96)	CI7(192)	0.173957276	ng
27	CI5(87)	CI5(87)	T	CI5(96)	CI7(192)	0.554767423	ng
28	CI5(99)	CI5(99)	T	CI5(96)	CI7(192)	0.187693487	ng
29	CI5(101)	CI5(101)	T	CI5(96)	CI7(192)	0.740833886	J1g
30	CI5(105)	CI5(105)	T	CI5(96)	CI7(192)	0.660391563	ng
31	CI5(110)	CI5(110)	T	CI5(96)	CI7(192)	0.991114455	g
32	C/5(114)	CI5(114)	T	CI5(96)	CI7(192)	0.249468861	ng
33	CI5(118)	CI5(118)	T	CI5(96)	CI7(192)	0.724437288	ng
34	CI5(119)	CI5(83)/CI5(119)	T	CI5(96)	C/7(192)		Ong
35	CI5(123)	CI5(123)	T	CI5(96)	CI7(192)	0.304088009	ng
36	CI5(126)	CI5(126)	T	CI5(96)	CI7(192)	0.218617352	ng
37	CI6(128)	CI6(128)	T	CI5(96)	CI7(192)	0.200727965	ng
38	CI6(138)	CI6(138)	T	CI5(96)	CI7(192)	0.545020176	ng
39	CI6(149)	CI6(149)	T	CI5(96)	CI7(192)	0.493449001	ng
40	CI6(151)	C/6(151)	T	CI5(96)	CI7(192)	0.253571392	ng
41	CI6(153)	CI6(153)/CL6(168)	T	CI5(96)	CI7(192)	0.706590699	ng
42	CI6(156)	CI6(156)	T	CI7(186)	CI7(192)	0.369477148	ng
43	CI6(157)	CI6(157)	T	CI7(186)	CI7(192)	0.811305994	ng
44	CI6(158)	CI6(158)	T	CI7(186)	CI7(192)	0.247984761	ng

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Project Test Code Summary Report

Lab Name: MS PestPCB

No:	Laboratory Analyte Name:	Report Name:	Type	RIS	SIS	DL	Units:
45	C16(167)	C16(167)	T	C17(186)	C17(192)	0.248054608	ng
46	C16(169)	C16(169)	T	C17(186)	C17(192)	0.256596197	ng
47	C17(170)	C17(170)	T	C17(186)	C17(192)	0.0454061219	ng
48	C17(177)	C17(177)	T	C17(186)	C17(192)	0.398554688	ng
49	C17(180)	C17(180)	T	C17(186)	C17(192)	0.507194161	ng
50	C17(183)	C17(183)	T	C17(186)	C17(192)	0.305361732	ng
51	C17(187)	C17(187)	T	C17(186)	C17(192)	0.220752048	ng
52	C17(189)	C17(189)	T	C17(186)	C17(192)	0.294424494	ng
53	C18(194)	C18(194)	T	C17(186)	C17(192)	0.220752048	ng
54	C18(195)	C18(195)	T	C17(186)	C17(192)	0.170444863	ng
55	C18(201)	C18(201)	T	C17(186)	C17(192)	0.252439625	ng
56	C18(203)	C18(203)	T	C17(186)	C17(192)	0.099196813	ng
57	C19(206)	C19(206)	T	C17(186)	C17(192)	0.0439376354	ng
58	LaC 1	LaC 1	T	C15(96)	C13(36)	6.146051056	ng
59	LaC 2	LaC 2	T	C15(96)	C13(36)	3.070195341	ng
60	LaC 3	LaC 3	T	C15(96)	C13(36)	7.847387048	ng
61	LaC 4	LaC 4	T	C15(96)	C17(192)	4.883211049	ng
62	LaC 5	LaC 5	T	C15(96)	C17(192)	11.82543948	ng
63	LaC 6	LaC 6	T	C17(186)	C17(192)	10.09582812	ng
64	LaC 7	LaC 7	T	C17(186)	C17(192)	2.277639157	ng
65	LaC 8	LaC 8	T	C17(186)	C17(192)	7.629287785	ng
66	LaC 9	LaC 9	T	C17(186)	C17(192)	2.780772945	ng
67	C110(209)	LaC 10	T	C17(186)	C17(192)		ng
68	C13(36)	C13(36)	SIS	C15(96)			ng
69	C17(192)	C17(192)	SIS	C17(186)			ng
70	C15(96)	C15(96)	RIS				ng
71	C17(186)	C17(186)	RIS				ng

Total Analytes: 70

Subtract Peaks:

None

Sum Peaks:

None

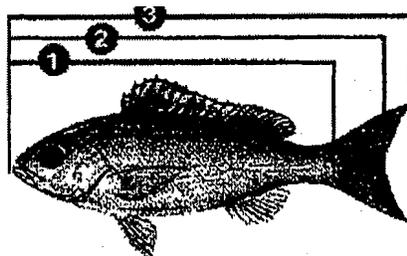
ATTACHMENT 2 FISH MEASUREMENT TECHNIQUES

1.3.2 Length

The fish length will be determined on a measuring board with a scale (mm) with a rigid head. The board will be used with a second measuring board prior to the fish. Before taking measurements of each fish, the measuring board will be visually inspected to ensure that the board is in good working order. The board will be rinsed with water between fish.

1. Place a measuring board on its right side with its head to the recorder's left.
2. Hold the head of the fish firmly against the head piece before measuring the fish.
3. Measure the total length to the nearest millimeter. Total length is defined as the length from the most anterior part of the fish to the tip of the longest fin ray.
4. Measure standard length to the nearest millimeter. Standard length is defined as the length of a fish from the front of the upper lip to the end of the vertebral column.
5. Record the fish length on the data sheet in the next fish number.

Exhibit 2. Diagram of Length Measurements



- (1) Standard
- (2) Fork Length
- (3)

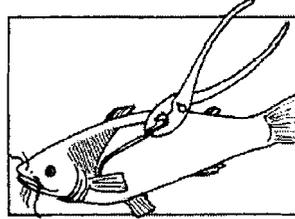
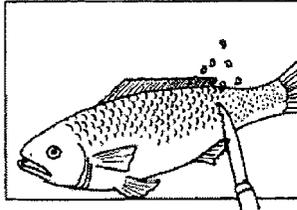
ATTACHMENT 3 FISH FILLETING TECHNIQUES

Scaled Fish

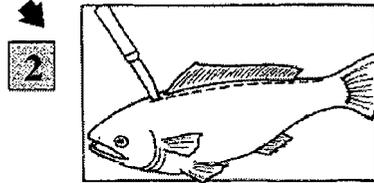
Scaleless Fish

After removing (by scraping with the edge of a rinsing the fish:

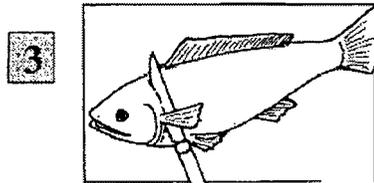
Grasp the skin at the base of the head (preferably pliers) and pull toward the tail.



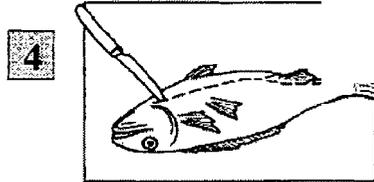
Note: This step applies only for catfish and other scaleless species.



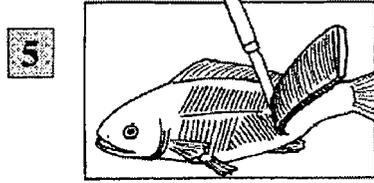
2 a shallow cut along the skin (on either side of the dorsal fin) from the top of the head to the base of the tail.



3 a cut behind the entire length of the gill cover through the skin and flesh to the bone.



4 Make a cut along the belly from the base of the tail to the gill cover. A single cut made from the gill cover to the anus then a cut is made on both sides of the anal fin. Do not cut into the gut cavity this may contaminate fillet tissues.



5 Remove the fillet.

ATTACHMENT 4 MEASUREMENT QUALITY OBJECTIVES

Element or Sample Type	Minimum Frequency	Acceptance Criteria	Corrective Action
ICAL, ICC, CCV, MS tuning criteria	Refer to SOP Montrose 002-05	Refer to SOP Montrose 002-05	Notify IEC. Sample(s) will be reanalyzed upon client direction.
IS area count	Refer to SOP Montrose 002-05	Refer to SOP Montrose 002-05	Notify IEC. Sample(s) will be reanalyzed upon client direction.
DDT breakdown	Refer to SOP Montrose 002-05	Refer to SOP Montrose 002-05	Refer to SOP Montrose 002-05
Standard Reference Material	One SRM with every batch (max 15 field samples)	%Difference values must be within ±15% of 95% confidence interval for the true or reference value ² . Up to 2 analyte values may be outside this range if the values are within ±25% of 95% C.I.	Notify IEC. Sample(s) will be reanalyzed upon client direction.
Method Blank	Every batch (max 15 field samples)	No analytes to exceed 3x MDL unless analyte not detected in associated sample(s) or analyte concentration > 10x blank value.	Notify IEC. Sample(s) will be reanalyzed upon client direction.
Laboratory Control Sample Recovery (%R)	Every batch (max 15 field samples)	%R' = 50% to 125%. (low-level spike may have some exceedences)	Notify IEC. Sample(s) will be reanalyzed upon client direction.
Matrix Spike Recovery	Every batch (max 15 field samples)	%R' = 50% to 125% if sample concentration is < 4X the matrix spike concentration.	Notify IEC. Sample(s) will be reanalyzed upon client direction.
Sample Duplicate Relative Percent Difference (RPD)	Every batch (max 15 field samples)	RPD ³ ≤ 30% if > 10x MDL for fillets; RPD ≤ 40% if > 10x MDL for whole body	Notify IEC. Sample(s) will be reanalyzed upon client direction.
Surrogate Recovery	Every sample (added prior to extraction)	% R=60% to 110%	Notify IEC. Sample(s) will be reanalyzed upon client direction.

1. See SOP Montrose 002-5 for equation.
2. SRM 1946

The following target analytes are certified: Pesticides 4,4'-DDT, 2,4'-DDD, 4,4'-DDD, 4,4'-DDE, dieldrin, a-chlordane, γ-chlordane, oxychlordane, cis-nonachlor, *trans-nonachlor*; PCB congeners 44, 49, 52, 66, 70, 74, 77,87,99, 101, 105, 110, 118, 126, 138, 149, 153/168, 156, 169/196, 170, 180, 183, 187,194, 195,206,209; Total extractable organics (TEO or % lipid)

$$RPD = \left(\frac{C1 - C2}{(C1 + C2)/2} \right) \times 100$$

3. RPD calculated as follows: where C1 is the larger of the duplicate results for a given analyte.

ATTACHMENT 5. DATA QUALIFIER DEFINITIONS



Glossary of Qualifiers

Flag: Application:

B		at	<10x the level	in the	blank.
D	Dilution Run.	Initial run	linear	of instrument.	
E		result greater than the	concentration level in the		
H		diluted out. Used When surrogate recover1 is affected	dilution of the		
J		the sample-specific Reporting	(RL).		
ME	Significant Matrix	-			
MI	Significant Matrix	-	could be determined or estimated.		
N	Control (OC) value is	accuracy or precision	(OO), but	the	criteria.
N	Control (OC) value	the	or precision	Quality	(DOO)
NA	Not				
T		(HT) exceeded.			
U	oot	at 3:1 signal:noise	. The sample-specific	detection	(MOL) reported.

ATTACHMENT 6 EDD SPECIFICATIONS

The Laboratory shall provide all analytical results in electronic form as well as hardcopy form. The electronic files must follow the structure and format defined in this article. Electronic deliverables shall match results reported in the hard copy and shall be received by MSRP along with the hard copy. Each electronic deliverable will be accompanied by a cover letter indicating which samples and respective sample delivery groups (SDGs) are included and the methods reported, and a hard copy of the laboratory report.

All electronic files will be audited by MSRP to determine if the specifications in this article have been followed. If a file format or structure does not meet specifications MSRP may request a complete resubmittal at no cost. Upon reviewing the electronic file, MSRP may also require a resubmittal based on inconsistencies (hereafter referred to as "error") with codes, spelling or missing information as specified in the Field Descriptions below. Resubmittals due to errors shall consist only of the error record(s) identified by MSRP at no cost. Resubmittals shall be received by MSRP no later than 5 working days from the time the Laboratory is notified by MSRP.

1.1 File Contents

Electronic deliverables shall contain all results reported on the hard copy of the laboratory report. The file shall include all analytical results if they are part of the method being reported, including:

- Environmental Samples
- Method Blanks
- Reference Materials
- Confirmation Results (for dual columns)
- Tentatively identified compounds (TICs)
- Matrix Spikes (MS)/Matrix Spike Duplicates (MSD)
- Laboratory control spikes (LCS) and laboratory control spike duplicates (LCSD)
- Field Duplicates/Replicates
- Surrogate Spikes
- Labeled Compounds
- Re-analyses
- Dilutions

1.2 Format

The file format shall be ASCII with a fixed length of 478 characters per record having the structure defined below. All fields identified in TABLE 1-1 must be included in the ASCII file in the exact order shown. Some fields, as identified below, may not be required to be populated and may be left blank. MSRP reserves the right to request limited modifications to the following structure at no cost to MSRP. Limited modifications include up to three additional fields and changes in field widths.

Table 1-1: ASCII File Fonnat

Field	Name	Type ¹	Width	Columns	Data Required ²
1	PROJECT	C	8	1-3	No
2	SAMPID	C	30	9-38	Yes
3	FLDBATCH	C	10	39-48	No
4	SAMPDATE	D	8	49-56	No
5	FIELDID	C	30	57-86	No
6	LABID	C	15	87-101	Yes
7	SDG	C	15	102-116	Yes
8	LABBATCH	C	15	117-131	Yes
9	ANALYTE	C	40	132-171	Yes
10	ANAORDER	C	3	172-174	No
11	VALUE	N	20	175-194	Yes
12	VALUESF	N	1	195	No
13	LABOUAL	C	10	196-205	Special
14	DVOUAL	C	4	206-209	No
15	DVOUALRC	C	10	210-219	No
16	UNITSCODE	C	10	220-229	Yes
17	DETLIMIT	N	20	230-249	Yes
18	DETLIMITSF	N	1	250	No
19	ANLGROUP	C	6	251-256	No
20	ANLMETHOD	C	20	257-276	Yes
21	MATTYPE	C	5	277-281	Yes
22	BASIS	C	3	282-284	No
23	EXTRDATE	D	8	285-292	Special
24	ANLDATE	D	8	293-300	Yes
25	ANLTIME	C	8	301-308	No
26	INST	C	10	309-318	No
27	EXTMETHOD	C	20	319-338	No
28	DILFACTOR	N	12	339-350	Yes
29	SAMPLEOTY	N	14	351-364	No
30	OTYUNITS	C	10	365-374	No
31	MOISTURE	N	10	375-384	No
32	RESORDER	C	3	385-387	Yes
33	OCTYPE1	C	4	388-391	Special
34	OCTYPE2	C	2	392-393	Special
35	OCTYPE3	C	3	394-396	Special
36	SPIKE	N	15	397-411	Special
37	RECOVERY	N	10	412-421	No
38	RPD	N	10	422-431	No
39	LOWLIMIT	N	7	432-438	No
40	UPPLIMIT	N	7	439-445	No
41	RPDLIMIT	N	5	446-450	No
42	ORIGRECNO	C	8	451-458	No
43	IMPFILE	C	12	459-470	No

44	IMPFILEDAT	D	8	471-478	No
<p>Notes:</p> <p>1Type column refers to the following data types:</p> <p>C Character, preferably left justified.</p> <p>N Numeric, no decimal defined. These fields must be fixed length in the ASCII file according to the specifications above, regardless of the number of digits or placement of the decimal. The decimal point can be present anywhere in the field (<i>i.e.</i>, 0.000001 or 100000.0) and is included in calculating the total field width.</p> <p>D Dates must be 8 characters long with the format MM/DDNY in the text file. If dates cannot be reported, 8 spaces must be present in the ASCII file or " / / " may be present.</p> <p>T Time must be 8 characters long in the format of HH:MM:SS (hours, minutes, and seconds).</p> <p>2Data Required column indicates the following:</p> <p>Yes The field <i>must</i> contain some information and a blank value is <i>not</i> acceptable.</p> <p>No The field does not require information and if left blank, is assumed to mean no information was supplied.</p> <p>Special A special case where the field may be left blank if appropriate, however, a blank field does <i>not</i> represent a lack of information, rather, it indicates some meaning (<i>i.e.</i>, a blank in LABQUAL indicates a detected result).</p>					

Field Descriptions:

1. **PROJECT:** The name of the project used to distinguish the data set from other data sets.
2. **SAMPID:** The sample identification number determined by MSRP. Every effort should be made by MSRP to ensure this identifier is unique. However, no modifications or additions to SAMPID in any form are acceptable unless specified by MSRP.
 QC samples created by the Laboratory from field samples (*e.g.*, matrix spikes and laboratory duplicates) must contain the exact SAMPID of the field sample. Other Laboratory QC samples (*e.g.*, blanks, blank spikes, and reference materials) must have unique sample identifiers which may be identical to the LABID below.
3. **FLDBATCH:** The field identification number determined by MSRP used to associate sampling event field QC samples.
4. **SAMPDATE:** Date the sample was collected.
5. **FIELDID:** The sample identification number determined by MSRP as reported on the chain-of-custody and on sample labels, or the laboratory QC sample identification.
 QC samples created by the Laboratory from field samples (*e.g.*, matrix spikes and laboratory duplicates) must contain the exact SAMPID of the field sample. Other Laboratory QC samples (*e.g.*, blanks, blank spikes, and reference materials) must have unique sample identifiers which may be identical to the LABID below.
6. **LABID:** The Laboratory internal identification number. MSRP assumes that the combination of the SDG and LABID field is sufficient to uniquely define either an environmental or QC sample; but may not be sufficient to distinguish reanalyses and dilutions.
7. **SDG:** The Sample Delivery Group number assigned by the Laboratory to identify a batch of samples. If the Laboratory's SDG batch includes multiple analytical batches or QC batches for a single method, the Laboratory must add additional alpha-numeric fields to define unique analysis and QC batches. This must be explained in the narrative with the electronic deliverable.
8. **LABBATCH:** The laboratory identification number used to associate laboratory generated QC samples.

9. **ANALYTE:** Analyte or parameter reported. CAS numbers may be reported in lieu of compound names. All compounds shall be reported in upper case.
10. **ANAORDER:** Usually reported using numeric order. Field designating the order in which the analytes are reported.
11. **VALUE:** Concentration, value, or result of the compound tested, reported to the correct number of significant figures. The method detection limit (MDL) will be reported for non-detect values. Only numbers are acceptable for this field.
In the case of spiked results, the VALUE will be the spiked sample result and will not be adjusted for the original sample results. If spiked compounds, such as surrogates, are diluted beyond detection, then the method detection limit (MDL) shall be reported in the VALUE field and a "U" added with other qualifiers in the LABQUAL field. A blank value is acceptable, indicating a non-spiked, non-reference material result.
12. **VALUESF:** The number of significant figures that should be reported for the VALUE field.
13. **LABQUAL:** Lab qualifiers are reported in this field. Please read the definition of 20. RESORDER also, since many common laboratory qualifiers actually belong in that field.
Qualifier codes may be used from either the *Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration*, Document OLM01.0 through revision OLM01.8 (U.S. EPA, August 1991) or *Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration*, Document ILM02.0 with revision ILM02.1 (U.S. EPA., September 1991). More than one qualifier may be used per record. If other qualifiers are used, then the Laboratory must include a typed list of the definitions of the codes with the electronics. The list must be present as both a paper copy and an electronic text file.
All nondetect results shall be reported with a "U" qualifier. The qualification "ND" for nondetect results is unacceptable. Blank values are acceptable and implied to mean a detected result. If a range will be reported (e.g., greater than 50) the symbol ">" shall be reported in this field.
14. **DVQUAL:** Data validation qualifiers are reported in this field.
15. **DVQUALRC:** Data validation qualifier codes are reported in this field.
16. **UNITSCODE:** The units of measure for each record will be reported in this field. A small "u" rather than a Greek mu (μ) MUST be used as an abbreviation for micro.
17. **DETLIMIT:** Used to report the Method Detection Limit (MDL). Non-detect results reported in the VALUE field should contain the MDL corrected for dilution, percent moisture, or related factors that affect the MDL.
18. **DETLIMITSF:** The number of significant figures that should be reported for the DETLIMIT field.
19. **ANLGROUP:** Field used to group results from various methods. For instance, an entry of 'METALS' may be entered to report results from methods SW-846 6010, SW-846 7041, and SW-846 7470.
20. **ANLMETHOD:** Indicate the analytical method used (e.g., SW-846 8240). Dissolved metals must be clearly identified versus total metals results. Toxicity characteristic leaching procedure (TCLP) results must be clearly identified as such while including the method number.
21. **MATTYPE:** Indicate one of the following for the medium analyzed: SOIL, SEDIMENT, SOLID (for solids that are not soil or sediment samples), WATER, NON-AQUS (for non-aqueous liquids), MIXTURE (this is only for samples that are mixed liquid and solid phases analyzed together) or AIR. If a sample or laboratory QC material does not match one of these, indicate with a code of "X" and notify MSRP in the cover letter.
22. **BASIS:** Indicate whether results are reported on a dry weight or wet weight basis, using the terms DRY or WET. If a sample or laboratory QC material does not match one of these, indicate with a code of "X" and notify MSRP in the cover letter.
23. **EXTRDATE:** Date the sample was extracted or prepared. If an extraction or preparation step is not applicable, then the field may be blank.
24. **ANLDATE:** Date the sample was analyzed.

25. **ANLTIME:** Time the sample was analyzed. This field is used to establish sample sequencing and is considered critical for MSRP to evaluate the potential for cross sample contamination. If this value is not readily available, but run order is, then please contact MSRP for a minor revision to the format.
26. **INST:** Instrument identifier
27. **EXTMETHOD:** Method of extraction or digestion. If an extraction is not performed a "NA" is acceptable.
28. **DILFACTOR:** The dilution factor. This should also reflect "effective" dilutions achieved by increasing or decreasing sample or extracting solvent volumes from standard amounts. That is, pre-concentration steps will result in a dilution factor of less than I; this is OK.
29. **SAMPLEQTY:** Quantity or weight of the sample used for analysis.
30. **QTYUNITS:** The units of measure for the quantity or weight of the sample used for analysis.
31. **MOISTURE:** Moisture content of soil samples, expressed as percent moisture.
32. **RESORDER:** Indicate the result order to identify valid "duplication" of results including re-analyses, dilutions, and confirmation samples. This field applies to all samples, including laboratory QC sample information. One of the following codes must be used for each record:

<u>Code</u>	<u>Definition</u>
PI	Primary result.
PITX or DXTX	Tentatively Identified Compound. The X denotes the TIC number (<i>i.e.</i> , T1 and T2) since a TIC compound (<i>e.g.</i> , "UNKNOWN") may be reported more than once for a sample/method.
RX	Re-analysis. The X represents the re-analysis order. For example, RI would indicate the first re-analysis run, R2 would indicate the second re-analysis run.
2C	Second column confirmation.
MS	GCIMS confirmation.
HP	HPLC confirmation.
DX	Dilution, where X represents the dilution order. For example, DI would indicate the first dilution run, D2 would indicate the second dilution run, and the original sample receives a P1.

33. **QCTYPE1:** This field is used to identify laboratory QC samples. A blank value is acceptable, indicating the record is not one of the sample types below. One of the following codes must be used to identify the laboratory QC sample type:

<u>Code</u>	<u>Definition</u>
RM	Reference material.
MB	Method blank.
MS	Matrix spike.
MSD	Matrix spike duplicate.
LCS	Laboratory control spike.
LCSD	Laboratory control spike duplicate.
DUP	Duplicate (Laboratory duplicates only; field duplicates will have a unique SAMPID).
SDL	Serial dilution.

34. **QCTYPE2:** This field is used to identify Field QC. A blank value is acceptable, indicating the record is not one of the sample types below. One of the following codes must be used to identify the Field QC sample type:

<u>Code</u>	<u>Definition</u>
FB	Field blank.
BB	Bottle blank.
EB	Equipment blank or rinsate.
TB	Trip blank.
FO	Field duplicate (if known).
FR	Field replicate (if known).

35. **QCTYPE3:** This field is used to identify analyte types, including tentatively identified compounds (TICs), surrogate compounds, internal standards (IS), and labeled compounds (LC). A blank value is acceptable, indicating the record is not one of the analyte types below. One of the following codes must be used to identify the analyte type:

<u>Code</u>	<u>Definition</u>
SUR	Surrogate result.
TIC	Tentatively identified compound.
IS	Internal standard.
LC	Labeled compound.

36. **SPIKE:** If added, this refers to the spike concentration or amount expected, for example 100 for 100 ug/kg. Units of measure are implied from the UNITSCOPE field.
37. **RECOVERY:** Percent (%) recovery. A blank value is acceptable, indicating a non-spiked, non-reference material result. This field is expected to be filled in for surrogates and labeled compounds as well as spiked QC samples and reference materials.
38. **RPD:** Relative percent difference. This field is expected to be filled in for field and laboratory duplicate, matrix spike duplicates, and laboratory control sample duplicates.
39. **LOWLIMIT:** Lower recovery control limit. This field is expected to be filled in for surrogates, QC samples and reference materials.
40. **UPPLIMIT:** Upper recovery control limit. This field is expected to be filled in for surrogates, QC samples and reference materials.
41. **RPDLIMIT:** Relative percent difference control limit. This field is expected to be filled in for field and laboratory duplicate, matrix spike duplicates, and laboratory control sample duplicates.
42. **ORIGRECNO:** Unique identifier field used in conjunction with IMPFILE and IMPFILEOAT to create unique records for all records in the database.
43. **IMPFILE:** Name of the import file used to create the database table.
44. **IMPFILEDAT:** File date of the import file used to create the database table.

1.3 Acceptance Criteria

1.3.1 Deviations from Specifications

It is the responsibility of the Laboratory to fully understand and meet the requirements of the electronic deliverable. The Laboratory should contact MSRP if clarification is needed. If the final deliverable package is determined to be of unacceptable quality, the package will be returned in part or entirety to the Laboratory to correct at no cost to MSRP.

If the Laboratory deems it necessary to assign a code or result to a record that does not match these specifications, the Laboratory must contact MSRP for approval prior to final submittal to MSRP. The Laboratory must also indicate any approved deviations in the cover letter.

Electronic files will be audited electronically to determine correctness based upon format and criteria described above. Files and individual records that do not meet these specifications (as modified by any approved deviations) must be corrected by the Laboratory, at no additional cost to MSRP.

1.3.2 Data Entry Verification

The Laboratory is fully responsible for creating an electronic deliverables package that is free of transcription errors (except those caused by poor writing on the chain-of-custody forms and/or on field container labels). The Laboratory is expected to perform 100% transcription error checking on all manually entered data, and to have thoroughly tested all computer programs used to generate the deliverable package. Errors caused by "buggy" computer programs and faulty data entry must be fixed within 5 days by the Laboratory at no cost to MSRP.

MSRP will review all deliverable packages for clerical and transcription errors. Random checks of an appropriate sample size will be performed to determine correspondence between hard (paper) copy and electronic results.